Original Article: Convenient Synthesis of 2,3-Diaryl-4-Thiazolidinones in Aqueous SDS Micelles

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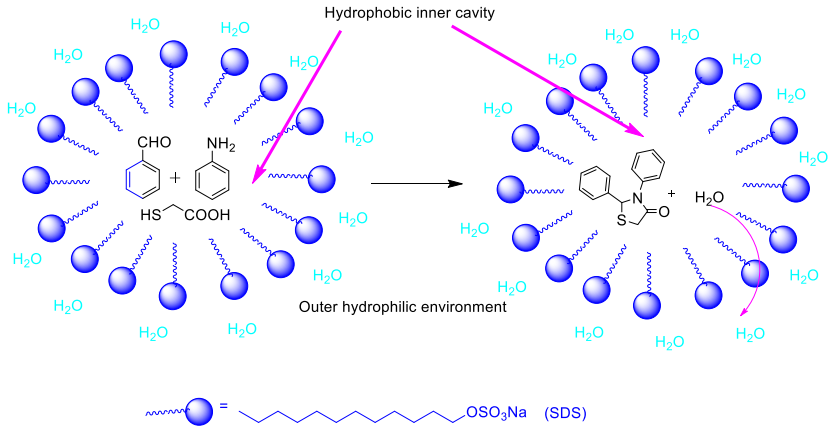
ABSTRACT

An efficient, rapid and green protocol has been developed for synthesis of 2,3-diaryl-4-thiazolidinones in aqueous micellar emulsion of anionic surfactant, sodium dodecyl sulphate (10 mol% SDS) at reflux condition. 11 examples leading to different 4-thiazolidinones are presented with a range of 77-85% of yields. Various substituted reactants are compatible with the developed procedure which shows versatility of the new synthetic method. The process is simple and high yielding.



Introduction

Thiazolidinones are receiving much importance and are extensively studied in recent years due to their synthetic and biological significance. They constitute an important group of heterocyclic compounds having valuable biological activities from pharmaceutical and agrochemical point of view [1]. The biological activities of thiazolidinones are considered to be associated with their capability to assume a

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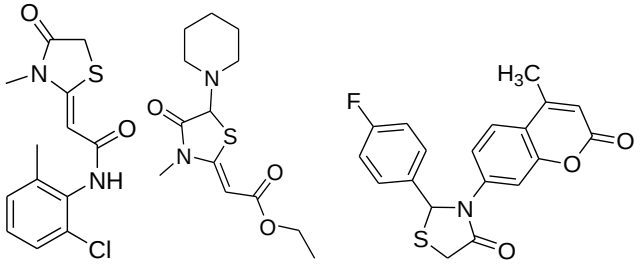
'butterfly like' conformation. Some of the interesting biological activities exhibited by thiazolidinone derivatives are anticancer [2] antimalarial [3], antitubercular [4], anti-histaminic [5], anti-convulsunt [6], antibacterial [7], antifungal [8], antiarrythmic [9], antioxidant [10], antagonist, anti-inflammatory [11], COX-1 inhibitor [12], anti-HIV [13] and entamoebahistolytica inhibitory [14]. Some thiazolidinone derivatives also act as hypoglycemic agents [15]. Following is a short review on therapeutical importance of 4-thiazolidinones.

Brown and Singh *et al.,* reviewed the synthesis, chemical reactivity and biological activity of 4-thiazolidinones [16]. Sala et al. synthesised 2,3-thiazolidin-4-one derivatives and tested for cytotoxic activity on human breast cancer cell lines [17]. Recently, Avdieiev et al. reported thiazolidinone derivatives as Bradykinin antagonists [18].

Thiazolidinone nucleus has been considered as a magic moiety (wonder nucleus); particularly, 4-thiazolidinone moiety is very versatile and has featured in many drugs such as Ralitoline **(A)** and Etozoline **(B)**. Several compounds with 4-thiazolidinone core structure have been found to be selectively effective against drug resistant cancer cells and induce cell death [19].



A **B** **C**

Figure1. Bioactive molecules containing thiazolidinone moiety

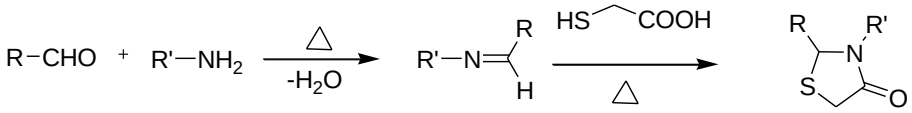
7-(2-Substituted phenylthiazolidinyl)-benzopyran-2-one derivatives have been prepared by Ronad et al., which are found to inhibit the growth of various bacterial and fungal strains [20]. The results showed that most of the compounds of the series exhibited good antibacterial and antifungal activities as compared with standard antibiotics Ciprofloxacin and Griseofulvin. Compound 2-(4-fluorophenyl)-3-(4-methyl-2-oxo-2H-chromen-7-yl)thiazolidin-4-one **(C)** is found to be the most active derivative with varying degree of inhibition as tested against bacteria *B. subtillis* and *S. aureus* as well as antifungal potency against *Aspergillus niger*.

Jain *et al.* reviewed the recent developments and biological activities of thiazolidinone derivatives [21]. Jadav et al. reported [22] thiazolidinone derivatives as inhibitors of chikungunya virus recently. Kothanahally et al. reported synthesis and antiproliferative effect of novel 4-thiazolidinones on human leukemic cells [23].

Reported methods for the synthesis of 4-thiazolidinones

In view of the biological/pharmacological significance of 4-thiazolidinone derivatives, substantial attention has been paid to the synthesis of this privileged class of heterocycles [24].

The main synthetic route to 1,3-thiazolidin-4-ones involves three components cyclocondensation of amines, carbonyl compounds and mercapto acetic acid [25]. The classical synthesis reported can be either a one-pot three-component condensation or a two-step process **(Scheme 1)**. The condensation initiated with the formation of an imine, which further invites nucleophilic addition of thioglycolic acid and finally leads to intramolecular cyclization on elimination of water [25].



Scheme 1. Synthetic route of 1,3-thiazolidin-4-ones

Thakare*et al.* reported three-component one pot silica gel promoted synthesis of 2,3-disubstituted 4-thiazolidinones. [26]. Recently, a biocatalytic method for the synthesis of 2,3-diaryl-4-thiazolidinones has been reported wherein *Saccharomyces cerevisiae* (baker's yeast) was used to catalyze the cyclocondensation of the aryl aldehydes, amines and thioglycolic acid [27]. In this method, the generation of solid waste and prolonged reaction time (40 h) are the major drawbacks. Bolognese and coworkers [28] prepared a range of 1,3-thiazolidin-4-one derivatives via microwave-assisted reaction between benzylidenes and mercaptoacetic acid in benzene at 30°C. Although the reaction is performed at lower temperature and completes faster, the use of hazardous solvent benzene is the main drawback of the protocol.

Along with these, there are several reports appeared in the literature, aiming at accelerating the one pot three component cyclocondensation leading to 4-thiazolidinones using catalysts like dicyclohexyldicarbodiimide [29], 1,1,3,3-tetramethyl uraniumhexaflurophosphate [30], zinc chloride [31], [bmim] activated fly ash [32], N-methyl pyridinium tosylate [33], silica chloride[34], Fe_3O_4@SiO_2nanoparticles [35] and ionic liquid immobilized on $FeNi_3$nanocatalyst[36]. The use of microwave heating [37], and polymer supported [38] systems has also been reported to obtain the target molecules.

The above-mentioned methodologies suffer from certain demerits such as use of carcinogenic solvents, prolonged reaction time, need of simultaneous removal of reaction water by Dean and Stark distillation system or by incorporating desiccant and need of inert and dry atmosphere to accelerate the cyclocondensation. The DCC mediated route has been found to be better. However, the separation of the byproduct, dicyclohexyl urea is tedious [29]. As the environmental issues are preferentially considered while developing any new synthetic method, it is highly desirable to think about the use of green procedures for the synthesis of value-added materials [39].

Water as a cheap and green solvent could be considered as an alternative reaction medium for organic processes [40]. Moreover, water with its unique physical and chemical properties shows selectivity and reactivity in reactions conducted in aqueous medium, which cannot be observed in organic solvents [41]. However, organic solvents are still used for most of the organic reactions due to insolubility of reactants. One way to overcome the solubility issue is using surface-active compounds (surfactants) which are able to form micelles or vesicular structures in aqueous reaction medium [42]. Use of micellar and vesicle-forming surfactants as catalysts is widespread and has been investigated in detail for different aqueous phase reactions [43]. However, micellar-aided organic reactions are still at their preliminary stages. Recently, ring-opening of epoxides with various nucleophiles [43] and Michael addition of amines and thiols to α-β-unsaturated ketones in micellar emulsion of sodium dodecyl sulfate (SDS) have been reported [44].

Scope and objectives

Considering all the above facts and in connection with previous efforts to modify synthetic routes in order to make them more environmentally acceptable [45], here the objective was set to develop an efficient, rapid and green protocol for one pot three componenetcyclocondensations of aryl aldehydes, aryl amines and thioglycolic acid in aqueous medium under relatively mild reaction conditions to obtain 4-thiazolidinones. To achieve these objectives, it was therefore planned to carry out the cyclocondensation using aqueous SDS micelles to accelerate the

reaction for obtaining efficiently high yield of 4-thiazolidinones.

Present work

In the present work an efficient, rapid and green protocol has been developed for synthesis of 2,3-diaryl-4-thiazolidinones in aqueous micellar emulsion of anionic surfactant, sodium dodecyl sulphate (SDS) at reflux condition.

Experimental Section

General

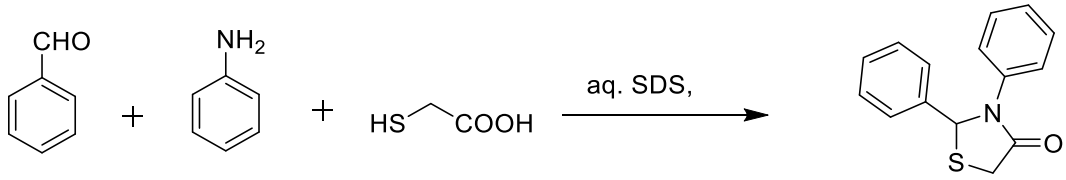
Melting points were determined by open capillary method and are uncorrected. Progress of the reaction was monitored by thin layer chromatography on MERK's silica plates. ^{1}H NMR and ^{13}C NMR spectra were recorded on Bruker Avance (500 MHz FTNMR) using TMS as an internal standard. Mass spectral data were determined by ESI-TOF (time of flight) mode on maXis impact 282001.00081 mass spectrometer. SDS (Sodium dodecyl sulphate) used was of Merck. All chemicals used were reagent grade and used without further purification.

General procedure for the synthesis of 2, 3-diaryl 4-thiazolidinones:

Aryl aldehydes (5 mmol), aryl amines (5 mmol) and thioglycolic acid (6 mmol) were mixed and stirred at 110°C in round bottomed flask to form homogenous mass. To this reaction mass 10 mol% of SDS was added with 10 mL water and further stirred at reflux condition. Progress of the reaction was monitored by TLC using ethyl acetate: pet ether (3:7) as a solvent system. After 50 minutes, the reaction mass was poured on ice water with stirring and to this then $NaHCO_3$ was added in portions till reaction mass become alkaline and then kept overnight, at room temperature. The whole reaction mass was then filtered, washed with plenty of water and dried under IR lamp. The crude product thus obtained was further purified by crystallization from absolute ethanol.

Results and Discussion

The investigations were started by examining the reaction of benzaladehyde (5 mmol), aniline (5 mmol) and thioglycolic acid (6 mmol) as a model reaction in aqueous micellar emulsion of SDS. It was noticed that the cyclocondensation did not run at RT even in aqueous SDS **(Table 1, entry 1)**. When the same reaction happened at elevated temperature, it was observed that reactants were converted in to corresponding cyclocondensed product, thiazolidinone; but the yield was not satisfactory **(Table 1, entries 2, 3)**. Hence the reaction temperature was further elevated to reflux and the yield of the desired cyclocondensed product was found to be increased to 85% **(Table 1, entry 4).**



Scheme 2. Specific reaction for synthesis of 2,3-diaryl-4-thiazolidinone

Table 1. Optimization for appropriate reaction temperature[a](**Scheme 2**)

Entry	Reaction temperature (°C)	Time (min.)	Yield(%)[b]
1	R T	50	nd
2	60	50	30
3	80	50	57
4	Reflux	50	85

[a]Reaction conditions: benzaldehyde (5 mmol), aniline (5 mmol), thioglycolic acid (6 mmol) SDS (20 mol% in 10 mL water).
[b]Isolated yields.

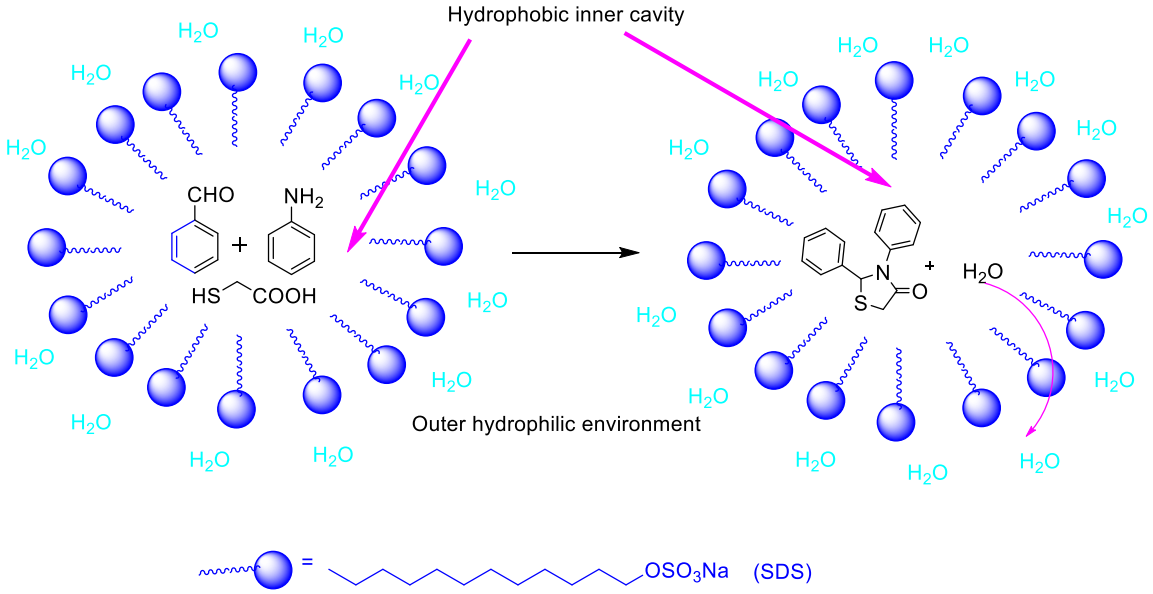
To optimize the appropriate amount of SDS, required for the efficient conversion of reactants into products, the model reaction was run with different amount of SDS, such as 2, 5, 10, 20 mol% at reflux condition **(Table 2)** and 10 mol% was found to be the appropriate amount essential for the conversion of reactants in to products. When the same reaction happened without SDS at similar reaction conditions, formation of cyclocondensed product was not detected even after 24 h **(Table 2 entry 5).**

Table 2. Optimization for appropriate amount of SDS[a] **(Scheme 2)**

Entry	SDS (mol%)	Time (min.)	Yield(%)[b]
1	2	50	Trace
2	5	50	64
3	10	50	85
4	20	50	85
5	-	50	nd[c]

[a]Reaction conditions: benzaldehyde (5 mmol), aniline (5 mmol), thioglycolic acid (6 mmol) in10 mL water.
[b]Isolated yields.
[c]Not detected, reaction time extended up to 24h.

SDS is an emulsifying agent and water acts as emulsifying medium. SDS, above its critical micellar concentration, forms colloidal particles in water and creates micelles as shown in **Scheme 3**. Thus, each generated micelle invites relatively nonpolar organic reactants, i.e. benzaldehyde, aniline and thioglycolic acid in its hydrophobic core. This therefore results in localized solublization of reactants in micelles, which are not soluble in water. The localized solubility of reactants in micelles, having their high concentration within these cells, is responsible for rate acceleration of the cyclocondensation. After conversion of reactants into product, thiazolidinone; the product then expels out from the inner core of micelles and the micellar inner core is again recharged with reactants.



Scheme 3. Proposed model for the synthesis of 2,3-diaryl-4-thiazolidinone in water in the presence of SDS

To test the generality of this reaction, a series of aromatic aldehydes were subjected with differently substituted aromatic amines and thioglycolic acid in aq. SDS emulsion as a reaction medium as well as catalyst at reflux temperature with stirring. Thiazolidinones with various substitutions were obtained in better to excellent yields **(Table 3, Scheme 4)**. Most of

the thiazolidinones synthesized by this protocol are known and having melting points in good agreement with those reported in the literature [34,35,46].

Spectral data of a representative compound of the series

Compound (4g): 2-(4-nitrophenyl)-3-*p*-tolylthiazolidin-4-one

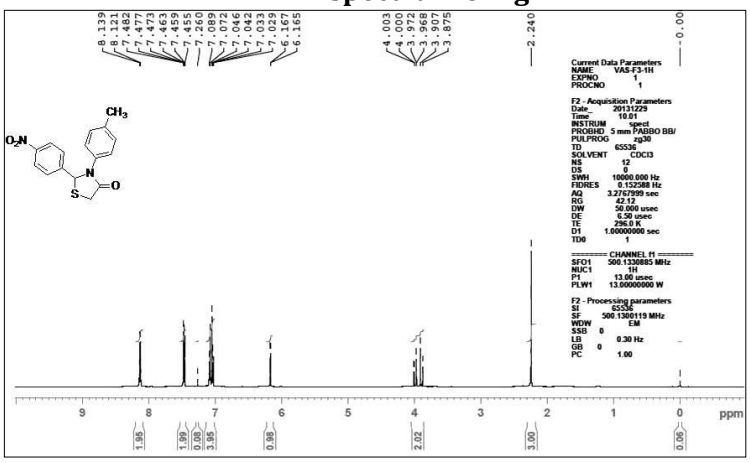
^{1}H-NMR (500 MHz, $CDCl_3$): δppm = 2.24 (s, 3H, CH_3), 3.9-4.0 (dd, 2H, Prochiral CH_2), 6.17 (s, 1H, CH), 7.03-7.09 (m, 4H, Ar-H), 7.47 (d, 2H, *J*=10 Hz, Ar-H) and 8.13 (d, 2H, *J* =10 Hz, Ar-H)

^{13}C-NMR (125 MHz, $CDCl_3$): δppm = 21.0, 33.4, 64.4, 124.3, 125.4, 128.0, 130.1, 134.3, 137.6, 146.9, 148.0 and 170.7.

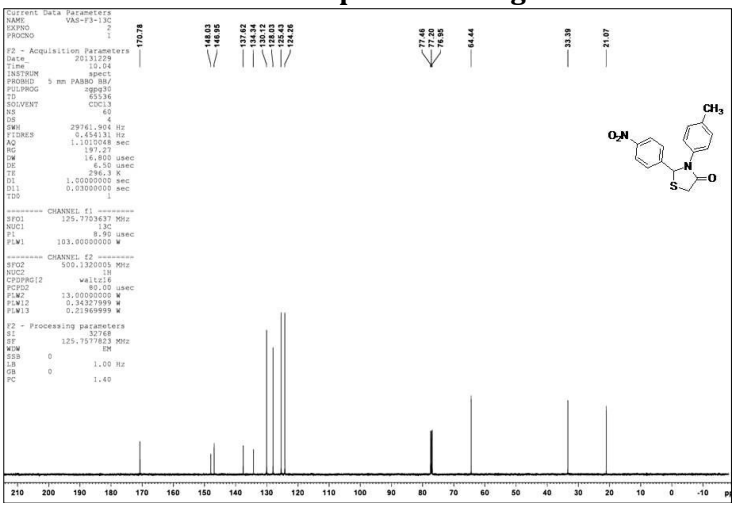
HR-ESI-MS (*m/z*):

Calculated for $C_{16}H_{14}N_2O_3S[M+ Na]^+$: 337.0617, found: 337.0613.

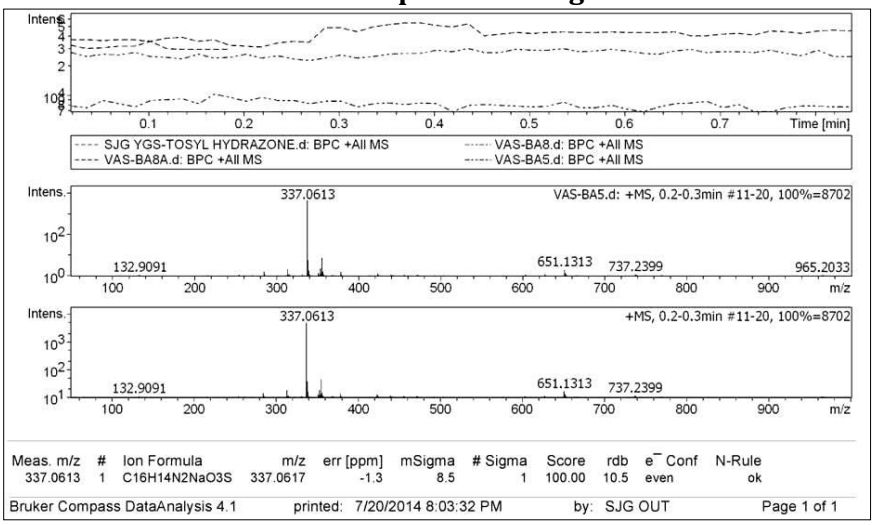
^{1}H-NMR spectrum of 4g

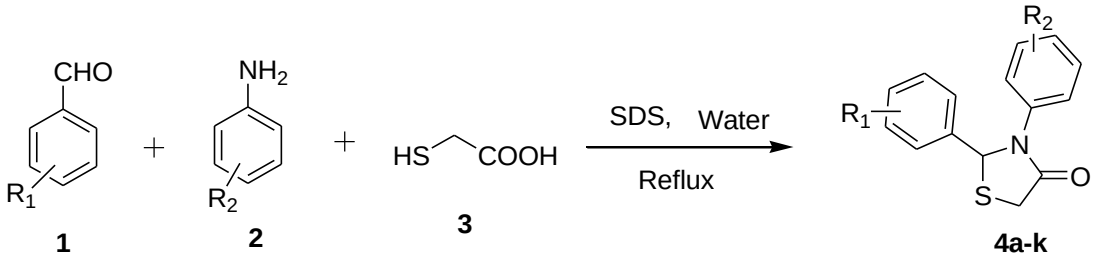


^{13}C-NMR spectrum of 4g



HR-MS spectrum of 4g





Scheme 4. Synthesis of 2,3-diaryl 4-thiazolidinones in aq. SDS micelles

Table 3. Synthesis of 2,3-diaryl 4-thiazolidinones in aq. SDS micelles[a] **(Scheme 4)**

Entry	R_1	R_2	Product	Yield(%)[b]	M. P. (°C)[c]
1	H	H	**4a**	85	131 – 132
2	4-OCH_3	H	**4b**	78	109 – 110
3	4-OH	H	**4c**	81	176 – 177
4	4-Cl	H	**4d**	80	133 – 134
5	H	4-CH_3	**4e**	81	110 – 111
6	4-Cl	4-CH_3	**4f**	78	161 – 163
7	4-NO_2	4-CH_3	**4g**	85	133 – 134
8	H	4-Cl	**4h**	80	110 – 111
9	4-OCH_3	4-Cl	**4i**	82	98 – 100
10	4-OH	4-Cl	**4j**	77	180 – 182
11	4-Cl	4-Cl	**4k**	79	123 – 125

[a]Reaction conditions: aryl aldehyde (5 mmol), aryl amine (5 mmol), thioglycolic acid (6 mmol) in SDS (10mol% in 10 mL water) at reflux condition.
[b]Isolated yields.
[c]The known thiazolidinones synthesized, by this method are having their melting points in good agreement with those reported in the literature [34,35,46].

Conclusion

In summary, a practical and convenient synthetic method in aqueous media using SDS as a surfactant catalyst (10 mol%) has been developed for the facile synthesis of 4-thiazolidinones. The operational simplicity, excellent yields of the products and high chemoselectivity are the main advantages of

this method. Furthermore, this procedure is rapid, economic and environmentally benign.

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Conflict of Interest

The authors do not have any conflict of interest regarding this research article.

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